PREPARATION AND CHARACTERISATION OF PHYSICOCHEMICAL PROPERTIES OF ALUMINIUM OXIDE (Al_2O_3) – WATER (H_2O) NANOFLUIDS USING TWO STEP METHODS

NUR ATIKAH BT ALI

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Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang

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ABSTRACT

Nanofluids is a kind of new engineering material which consisting of solid nanoparticles with size typically of 1-100nm by dispersing it into based fluids. This research presents the preparation of Aluminium Oxide (Al_2O_3) nanofluids by using two-step methods. This nanofluids was prepared by dispersing Al_2O_3 nanoparticles (<50nm) in distilled water based fluids with the addition of calculated amount of Sodium Dodecylbenzene Sulphonate (SDBS) as a surfactant and absence of SDBS surfactant. The nanofluids were sonicated with ultrasonic vibrator at power of 700 Watt and frequency of 40k Hz for 15 minutes. It was tested that with the presence of surfactant in the nanofluids, the nanofluids was stable for 2 weeks. Absence of surfactant cause the small amount of particle aggregates had settled out of suspension after 1 day. Stabilization of the Al_2O_3 nanofluids with and without surfactant can be obtained with Transmission Electron Microscopy (TEM). Distilled water based fluids display Newtonian behavior but it transform to non-Newtonian fluid with the addition of Al_2O_3 nanoparticles. Viscosity of the Al_2O_3 nanofluids was measured both as a function of Al_2O_3 nanoparticles volume fraction and temperature between 30^o C and 60^oC using Brookfield DV- II Pro Viscometer and results showed that no anomolous effects of viscosity with increasing of temperatures. The range of the viscosity from 30^o C and 60^oC is between 4.05 cP to 2.55 cP and viscosity of nanofluids increase with the Al_2O_3 nanoparticles volume concentration. The result then was compared with the previous researcher's experimental data. Thermal conductivity of the Al_2O_3 nanofluids water based was predicted using regression equation then compared it with previous experimental data.

ABSTRAK

Cecair nano adalah bahan kejuruteraan baru dimana ia terdiri daripada zarah-zarah nano yang bersaiz antara 1 – 100nm dengan melarutkan ia didalam cecair asas. Kajian ini adalah mengenai penyediaan cecair nano Alumina (Al_2O_3) menggunakan kaedah langkah ke-2. Cecair nano ini disediakan dengan melarutkan serbuk Alumina yang bersaiz < 50nm ke dalam air suling sebagai cecair asas dan ditambahkan sebilangan Sodium Dodecylbenzene Sulphonate SDBS sebagai bahan pengurai dengan kadar yang ditetapkan. Setelah itu, proses sonikasi pada kuasa 750 Watt dan frekuensi 40 kHz dengan adanya sonikasi gegar dilakukan pada cecair nano Alumina selama 15 minit. Dengan hadirnya SDBS sebagai bahan pengurai, dalam cecair nano, ia menunjukkan cecair nano Alumina itu stabil dan tiada mendapan berlaku selama 2 minggu. Tanpa kehadiran SDBS itu, ia menyebabkan sebilangan kecil serbuk Alumina itu termendap dalam tempoh satu hari dan menunjukkan ia tidak stabil. TEM digunakan untuk memperlihatkan kestabilan imej kedua-dua nano itu. Seperti yang diketahui, air suling mengandungi cirri-ciri cecair yang bersifat Newtonian tetapi bertukar kepada cirri-ciri Non-Newtonian dengan penambahan serbuk Alumina. Kepekatan cecair nano Alumina diukur dengan pengaruh bilangan serbuk nano Alumina dan suhu diantara 30 – 60^{o}C menggunakan Brookfield DV- II Pro Viscometer. Keputusan menunjukkan tiada perubahan yang ketara terjadi pada kepekatan cecair nano dengan peningkatan suhu. Kepekatan yang diperolehi adalah antara 4.05cP – 2.55cP dan bertambah dengan bertambahnya jumlah serbuk nano Alumina. Setelah itu, perbandingan diantara keputusan yang diperolehi dibandingkan dengan keputusan kajian yang pernah dilakukan oleh para kaji sebelum ini. Pengalir termal jugak ditentukan nilainya dengan menggunakan persamaan matematik yang telah direka olah para kaji.

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LIST OF SYMBOLS

Ag	Argentum
Al	Aluminium
Al_2O_3	Aluminium Oxide
$Al_{70}Cu_{30}$	Alloyed nanoparticles
AIN,SiN	Nitride Ceramics
Au	Gold
CB	Columbium
$C_{12}H_{25}C_6H_4SO_3Na$	Sodium dodecylbenzene Sulphonate
cP	centipoises
Cu	Copper
CuO	Copper Oxide
$CuSO_4.5H_2O$	Copper (II) Sulfate Pentahydrate
d_p	particle diameter
EG	Ethylene Glycol
EO	Engine Oil
Fe	Ferum
Fe_2O_4	Magnetic Oxide
$HClO_4$	Perchloric acid
H_2O	Water

$NaH_2PO_2.H_2O$	Sodium hypophosphite
nm	nano-meter
SiC	Silicon Carbide
T_b	Bulk temperature
TiO_2	Titanum Oxide
µm	micro-meter
ϕ	volume fraction

LIST OF ABBREVIATIONS

AN	Alkylated Naphthalenes
API	Air Pollutant Index
BSE	Backscattered Electrons
CVD	Chemical Vapor Deposition
DI	Distilled
FKKSA	Fakulti Kejuruteraan Kimia Sumber Asli
IGC	Inert Gas Condensation
PAG	Poly Alkylene Glycols
PAO	Poly Alpha Olefins
PE	Primary Electrons
PVD	Physical Vapor Deposition
RPM	Revolution per Minute
SANSS	Submerged Arc Nanoparticles Synthesis System
SDBS	Sodium Dodecylbenzene Sulphonate
SE	Secondary Electrons
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
UMP	Universiti Malaysia Pahang
UPM	Universiti Pertanian Malaysia

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Rapid increase in energy demand of worldwide for more efficient energy storage materials and inherently low thermal conductivity engender the strong need in many industrial field to intensifying heat transfer process and reducing energy loss by developing advanced heat transfer fluid with high thermal conductivities than are presently available. Beside than that, due to the serious *terawatt challenge while mitigating carbon emission and global warming* (Smalley, 2005) has become the major priority to securing clean energy of the countries.

Basically, heat must be transferred either to input energy into a system or to remove the energy produced in a system. Therefore, thermal conductivity is one of the important requirements of enhancement of energy heat transfer in industries such as transportation industry, microelectronics, industrial cooling system and thermal management, industrial heat exchanger coolants, electronic coolant and etc. For the transport of energy system like coolant, thermal conductivity is needed in order to preventing the overheating cooling. Further than that, thermal conductivity also needed to maintain the desired performance and reliability of a wide variety of product like computer, car engines, power electronics and laser or x-rays. Therefore, recently there were a lot of investigation and researches for improving the heat transfer characteristic have been constrained because traditional heat transfer fluids used in today's thermal management systems such as water, oils, ethylene glycol have inherently poor thermal conductivities, orders of magnitude smaller than those of most solids (Das et al., 2008).

Due to the development of nanoscience and nanotechnology field, many researches and investigation on nanofluids have been carried out actively whereby nanofluids is a mixture consisting of solid nanoparticles with size typically of 1-100nm (Zhu et al., 2009) and fibers dispersed in a liquid which can be made from chemical stable metal such as metallic and nonmetallic solid which have high thermal conductivity and stability than those of the conventional heat transfer fluid or the suspensions of micro-sized particles. In order for it to dispersed in base fluid like distilled and de-ionized water, engine oil, acetone, and ethylene glycol , the thermal conductivity should be high ($k_{liquid} <<< k_{solid}$). Therefore, the determination of nanofluids thermal conductivity has great importance whereby it can produce more stable suspension to reduce the coagulation of nanoparticles in the nanofluid rather than enhanced the problem encountered like clogging in slurries; erosions and sedimentation beside it give opportunity in enhancement of energy heat transfer. Hence, to formulating stable nanofluid with controlled properties like thermal conductivity, viscosity, and wettability for heat transfer application (Wen et al., 2009). It is also depends on the properties of the base fluid and the dispersed phase, particle concentration, size and morphology.

Numerous investigations and researches on colloidal dispersions also have been conducted by the researchers in view of particles motion analysis in various flow conditions and sedimentation characteristic studies on suspended nanoparticles in base fluids (Yujin Hwang, 2008). Beside than that,by studying the properties of surfactants like laurate salts,thiols and Oleic acid as it been added to nanofluids as an additive was known to be very effective to homogenously disperse nanoparticles in the base fluids and reduce the particles agglomeration due to van der Waals forces of attraction (Fendler JH, 2001).

The requirement of nanofluids formulations nowadays are very demanding compare to conventional colloids. Therefore, widely investigation and researches have been focus on nanofluids formulation whereby there were two methods can be generally used to produced nanofluids which are single step method known as the bottom up approach through simultaneous production and dispersion of nanoparticles and second method are the two-step method which known as the top down method through size reduction.

1.2 Nanofluids

Nanofluids are engineered colloidal suspensions of nanoparticles in base fluids. In general the size of these nanoparticles varies from 1-100nm in size. The type of nanoparticles used is directly dependent on the enhancement of a required property of the base fluid. All physical mechanisms have a critical length scale, below in which the physical properties of materials are changed. Therefore, particles < 100nm exhibits properties that are considerably different from those of conventional solids. Fundamentally, it can be concluded that studies on metallic nanofluids have opened a new horizon with highly enhanced thermal conductivity with low particle volume fraction. In the previous research Zhu et al. (2009) develop a novel two step methods for preparation of Aluminium Oxide (Al_2O_3) nanofluids by dispersing Aluminium Oxide nanoparticles into water based with and without presence of surfactant. The particle size distribution shows better dispersion behavior in the suspension with the addition of surfactant.

Hence, the Aluminium Oxide nanofluids can be prepare by dispersing directly the nanopowders to the water base fluid and sonicated it at required vibration time and the others affect parameters.

1.3 Research Objective

The aim of this research is to study the characterization of physicochemical of novel two step method for preparation of Aluminium Oxide water based nanofluids. Hence the objective of this research is:

i. To prepared Aluminium Oxide nanofluids using water based.
ii. To obtain stabilization of the Aluminium Oxide water based nanofluids by considering the surfactant used.
iii. To obtain the viscosity of the nanofluids at different ranging of temperature at different volume concentration of Aluminium Oxide nanoparticles.

1.4 Problem statement

The problem statement of this research is:

1. To investigated the rheological characteristics (viscosity) of the nanofluids over temperatures ranging from 30^0C to 60^oC by considering the volume concentration of Aluminium Oxide nanoparticles.

2. To estimate the thermal conductivity and viscosity of the Aluminium Oxide nanofluids by using regression equations and compared it with previous experimental results.

1.5 Scopes of research

The scope research for the preparation of Aluminium Oxide water based nanofluids is to investigate the rheological behaviour of these nanofluids over different ranging of temperature by considering the volume concentration of Aluminium Oxide nanoparticles used. Determination of viscosity of the nanofluids is essential to establishing adequate pumping power in the engineering application as well as the convective heat transfer coefficients. Apart from that, from the determination of viscosity at different temperature, non-newtonian properties of the nanofuids can be determined also. Estimation of thermal conductivity using regressions equation is to be applied and comparison with the experimental data is conduct.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The greatly increasing thermal load in microelectronics, high powered automobiles, the need for high performance heating and cooling fluids which increasing year by year made the thermal conductivity of these fluid plays a vital role in the enhancement of energy heat transfer. In order to improve the thermal properties of energy transmission fluid, there were an effective way to improving it by suspending small solid particles of mono or nano-size in the fluid because the thermal conductivities obtain when the small solid particles of mono (µm) and nano (nm) suspended in the fluid is greater than the common fluid. Hence, the novel concept of nanofluids whereby it is a heat transfer fluid containing suspension of nanoparticles (Wu et al. 2009) has been purposed in order to improve the energy heat transfer fluid including transportation industry, electronics cooling, industrial cooling system and etc.

2.2 Type of nanofluids

Nanofluid is a solid-liquid composite material which consists of 1-100nm with size of nanoparticles of nanofibers which will be added to the base fluid in order to obtain the thermal conductivity. There were two type material that can be used to prepare the nanofluid which is: (1) metallic solid and (2) nonmetallic solid.

2.2.1 Metallic solid and non metallic solid

As a theoretically, all solid nanoparticles with high thermal conductivity can be used as additives of nanofluids. Metallic solid or particles such as Cu, Al, Fe, Au and Ag (Li et al. 2009). Same as metallic particles, nonmetallic solid also can be an additives of nanofluid in enhancement of thermal conductivities in energy transfer. There were Al_2O_3, CuO, Fe_2O_4, TiO_2, SiC, carbon naotube and nanodroplet (Li et al. 2009). The thermal conductivities of each nonmetallic particle were shown in **Table 2.2.1.** Generally the thermal conductivity of the metallic solid is much higher than non metallic solid.

Table 2.2.1: Thermal Conductivity of additives and base fluids used in nanofluids

Material		**Thermal Conductivity, (W/m^2.K)**
Metallic solids	Cu	401
	Al	237
	Ag	428
	Au	318
	Fe	83.5
Non-Metallic solids	Al_2O_3	40
	CuO	76.5
	Si	148
	SiC	270
	CNTs	~3000(MWCNTs) ~ 6000 (SWCNTs)
	BNNTs	260 ~ 600

2.3 Preparations of Nanofluids

Preparation of nanofluids is the first key step in experimental studies by applying nanophase particles to improve the thermal conductivity of the nanofluids. The nanofluid does not simply refer to a liquid-solid mixture. There were some special requirements are necessary such as even suspension, stable suspension, durable suspension, low agglomeration of particles and no chemical change of the fluid (Xuan et al. , 2000)

As a general : (1) to change the pH value of suspensions ; (2) to use surface activators or disperstant ; (3) to use ultrasonic vibration are effective methods use for preparation of suspensions (Amrollahi et al., 2007). All these technique aim at changing the surface properties of suspended particles and suppressing formation of particles cluster in order to obtain stable suspensions (Xuan et al., 2000). There is mainly two techniques used to produces nanofluids : single steps methods and two-steps methods.

2.3.1 Single step methods

The single-step method is a process combining the preparation of nanoparticles with the synthesis of nanofluids, for which the nanoparticles are directly prepared by physical vapor deposition (PVD) technique or liquid chemical method. In this method the processes of drying, storage, transportation, and dispersion of nanoparticles are avoided, so the agglomeration of nanoparticles is minimized and the stability of fluids is increased. But a disadvantage of this method is that only low vapor pressure fluids are compatible with the process. This limits the application of the method (Li et al., 2009).

In the previous researches, Eastman et al. (2007) has used a one-step physical method to prepare nanofluids, in which Cu vapor was directly condensed into nanoparticles by contact with a flowing low vapor pressure liquid (ethylene glycol). Meanwhile Liu et al. (2006) synthesized nanofluids containing Cu nanoparticles in water through chemical reduction method for the first time. Lo et al. (2005) prepared copper dioxide nanofluids by a single-step method called SANSS. The established SANSS demonstrated to be effective in avoiding particle aggregation and producing uniformly distributed and well-controlled size of CuO nanoparticles dispersed in a deionized water suspension. Zhu et al. (2004) presented a novel one-step chemical method for preparing copper nanofluids by reducing $CuSO_4 \cdot 5H_2O$ with $NaH_2PO_2 \cdot H_2O$ in ethylene glycol under microwave irradiation. Non-agglomerated and stably suspended Cu nanofluids were obtained.

2.3.2 Two-step methods

The two-step method for preparing nanofluids is a process by dispersing nanoparticles into base liquids. Nanoparticles, nanofibers or nanotubes used in this method are first produced as a dry powder by inert gas condensation, chemical vapor deposition, mechanical alloying or other suitable techniques, and the nanosized powder is then dispersed into a fluid in a second processing step. This step-by-step method isolates the preparation of the nanofluids from the preparation of nanoparticles. As a result, agglomeration of nanoparticles may take place in both steps, especially in the process of drying, storage, and transportation of nanoparticles. The agglomeration will not only result in the settlement and clogging of microchannels, but also decrease the thermal conductivity. Simple techniques such as ultrasonic agitation or the addition of surfactants to the fluids are often used to minimize particle aggregation and improve dispersion behavior. Since nanopowder synthesis techniques have already been scaled up to industrial production levels by several companies, there are potential economic advantages in using two-step synthesis methods that rely on the use of such powders. But an important problem that needs to be solved is the stabilization of the suspension prepared (Li et al., 2009).

In the previous researches, Hong et al. (2005) prepared Fe nanofluids by dispersing Fe nanocrystalline powder in ethylene glycol by a two-step procedure. The Fe nanoparticles with a mean size of 10 nm were synthesized by a chemical vapor condensation process. Meanwhile, Xuan et al. (2000) prepared Cu/H_2O, Cu/oil nanofluids by two-step method. In order to avoid nanoparticle aggregation, surfactants and ultrasonic agitation were employed. Xie et al. (2002) prepared Al_2O_3/H_2O, Al_2O_3/EG, Al_2O_3/PO nanofluids by two-step method, and intensive ultrasonication and magnetic force agitation were employed to avoid nanoparticle aggregation.

2.4 Base fluids

To be able to formulate and use type of base fluid to be added with nanoparticles, the properties of the base fluids have to be well known. Base fluid properties that will influence the formulation could divide into two groups which are physical and chemical properties. In this study, properties from all of these groups are investigated to improve the understanding on their influence on base fluid overall performance whereby could divided into different groups, mineral, synthetic, or ester, and are classified in various ways. (Petterrson., 2005).

Researchers have also tried to increase the thermal conductivities of base fluid by suspending micro or nano-size solid particles in fluids since the thermal conductivity of solid is typically higher than that of liquids (Wang et al. 2007). Apart from that, liquid lubricants or based fluids may be characterized in many different ways. One of the most common ways is by the type of based fluids used. Following are the common types:

1. Water
2. Mineral oil
3. Vegetable Oil (natural oil)
4. Synthetic oils
5. Others

2.4.1 Water

Water is the chemical substance with chemical formula H_2O: one molecule of water has two hydrogen atoms covalently bonded to a single oxygen atom. It can be used on its own or as a major component in combination with one of the other base oils. Normally, water and steam are used as heat transfer fluids in diverse heat exchange systems, due to its availability and high heat capacity, both as a coolant and for heating. Beside than that, Water has the second highest molar specific heat capacity of any known substance, after ammonia, as well as a high heat of vaporization ($40.65\ kJ \cdot mol^{-1}$), both of which are a result of the extensive hydrogen bonding between its molecules. Therefore, water is suitable for the act as a heat transfer fluids and used in power generation, hydroelectricity and another heat transfer application. There was a lot type of water such as;

1. Tap water
2. Distilled water
3. De-ionized water

This type of water normally is used as a conventional fluid in heat transfer application due to their chemical and physical properties.

2.4.2 Mineral oil

Mineral oil or mostly known as liquid petroleum is a liquid by-product of the distillation of petroleum to produce gasoline and other petroleum based products from crude oil. Normally, mineral oil is transparent, colorless oil composed mainly of alkanes and cyclic paraffin. Furthermore, mineral oil is a substance of relative low value and available in light and heavy grades. There are three basic classes of refined mineral oils;

1. Paraffinic oils, based on n-alkanes
2. Naphthenic oils, based on cycloalkanes
3. Aromatic oils, based on aromatic hydrocarbons